# Local Plasticity in Al(0.5wt% Cu) Membranes Revealed by Scanning X-Ray Microdiffraction (µSXRD)

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## **INTRODUCTION**

While polycrystalline materials are ubiquitous in the industry of metal thin films, the understanding of polycrystal plasticity still constitutes a theoretical challenge. The problem arises from the fact that grain boundaries introduce a factor of complexity in plastic deformation that can not be adequately studied by conventional averaging techniques. On the other hand, transmission electron microscopy techniques, besides probing too fine a length scale to be statistically significant, cannot usually measure strain/stress of the material without disturbing its initial state (thinning). We have shown that the white beam  $\mu$ SXRD technique [1,2], developed at the ALS on beamline 7.3.3. is capable of measuring orientation and strain/stress variations in the correct mesoscale length scale without the need of any sample preparation which can alter the microstructure or true stress state of the material under investigation. In one of our study [3], we showed significant inter- as well as intragranular stress variations in polycrystalline Al thin films sputter deposited on a silicon wafer, in apparent contradiction with the nearly isotropically elastic properties of aluminum. The present study on a thin Al membrane, shows that grain-to-grain interactions play a prominent role in the understanding of the local stress distribution and must be considered as key parameters in the modeling of polycrystal plasticity.

#### **EXPERIMENTAL**

The sample is a Al(0.5 wt.% Cu) film deposited at 400 °C on a 200 nm thick  $SiN_x$  membrane on a Si frame and is used for in-situ bulge test experiment [4] allowing to apply stress to the film by differential pressure bulging. The aspect ratio of 1:6 of the membrane ensures a macroscopically uniaxial stress across the narrow dimension of the film.

 $\mu SXRD$  data were collected by scanning the same 15x15  $\mu m$  area of the film while differential pressure ranging from 0 to 250 Torrs were applied. At each step of the scans (step size of 0.5  $\mu m$ ), a white beam Laue pattern was collected, from which grain orientation and deviatoric stress tensor is deduced [1,2].

### **RESULTS**

Even before applying any differential pressure, the residual stress in the film appears in our  $\mu SXRD$  experiments to be strongly non-inhomogeneous with local variations reaching 100 MPa. The variations are both inter and intragranular. In particular shear stresses at grain boundaries can be significant. The stress state at room temperature is primarily a result of cool down from the 400 °C deposition temperature due to the difference in thermal expansion coefficients between

the film and the substrate. A wide distribution in stress implies a wide distribution in yield stresses, and can be explained by a grain size effect. Dislocation motions are limited by grain boundaries and the yield stress is on average inversely proportional to the grain size [5]. However a distribution of yield stresses due to difference in grain size or orientation cannot solely account for all aspects of the stress distribution. Our data indicate that the actual stress in the film is the result of complex interactions between different grains while experiencing a macroscopic applied stress. In particular, the stress state of a given grain is strongly influenced by those of its immediate neighbors as schematized in Fig. 1. A high yield stress area (small grains) and a low yield stress area (big grains) are sandwiched between two boundary grains. When an external constraint is applied, a shear stress appears at the boundary between the two different yield stress areas and a stress gradient appears in the boundary grains.

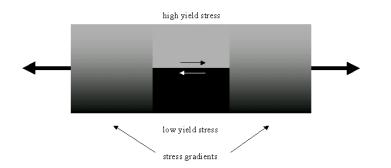


Fig. 1.- Schematic behavior of two grains with two different yield stresses sandwiched between two boundaries grains as an external force is applied.

Both effects are observed in the experimental data (Fig. 2). Grain B act as a high yield stress grain while grain C has a low yield stress. Shear stress is observed at the boundary between grain B and C (see  $\sigma_{xy}$  distribution) while a bottom to top stress gradient is evident in grain A (see  $\sigma'_{xx}$  and  $\sigma'_{zz}$  distributions).

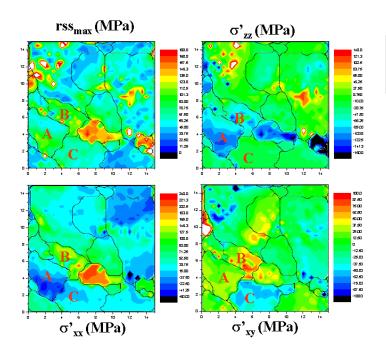


Fig. 2.- Resolved shear stress, deviatoric stress and shear stress as measured by  $\mu SXRD$  on an Al membrane under stress.

The active glide system in a given grain can be inferred by using the maximum resolved shear stress criterion (which is calculated from the deviatoric stresses considering the 12 {111}/<110>glide systems for fcc crystals). Our in-situ bulge test experiment clearly shows

changes in the active glide systems while the film experiences increasing applied stress during the in-situ bulge test experiment. In particular we notice that changes in the glide system can occur in part of a given grain under the influence of the changing stress state and shape of the neighboring grains. Therefore, during plastic deformation in polycrystals, large grains behave more like a collection of small grains (subgrains) rather than single crystals. This concept of an effective grain size where the material deforms uniformly, and which can be smaller than the physical grain size, is a direct consequence of the grain-to-grain interactions during deformation.

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